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### ANALYSIS OF THERMAL TREATMENTS FOR THE 7075 & 7091 ALUMINUM ALLOYS

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ABSTRACT (Continue on reverse side if necessary and id		
Retrogression and re-aging (RRA) is a therm	al treatment applied to 7000 series aluminum alloys	
of the T-6 temper. In the present work, 707	acking resistance while maintaining the peak strength 5 and 7091 were retrogressed at temperatures betwee	
160°C and 240°C and aged for 24 hours at 1	120 <sup>4</sup> C. The hardness and conductivity data obtained	
were compared to and contrasted with the a		
	ctive at retrogression temperatures lower than prev-	
iously reported and that retrogression appea	rs to be kinetically dominated (i.e. data fits a first	

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order dissolution model). The aforementioned results should enable the RRA process to be expanded to thick plates.					

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### INTRODUCTION

The 7000 Series (Al-Zn-Mg-Cu) aluminum alloys have been used in airframe structures for the last 25-30 years. These alloys provide high strength and stiffness, but they are prone to exfoliation and stress corrosion cracking (SCC), particularly when aged to the peak strength condition (T6). The resistance to SCC and exfoliation can be increased by over-aging to T76 and T73, although there is a loss of approximately 10-15% strength.

The 7091 alloy is a high strength powder metallurgy alloy that is age hardenable and has application similar to 7075. The mechanical and physical properties reported show that 7091 is superior to comparable ingot metallurgy alloys. Since 7091 is very close to 7075 in composition it was included in the RRA investigation. This paper investigates a retrogression and re-aging process of a 7075 ingot alloy and a 7091 power alloy.

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There have been a number of references to retrogression and re-aging RRA (References 1-5). This treatment was applied to the 7075 alloy in the T651 condition. It consists of heating in the temperature range 200-280°C for short time periods followed by re-aging using the conditions of a T6 age. The various T6 and T7 tempers are given in Table 1 (references 6-9). During retrogression, hardness/strength falls very rapidly during the first few minutes, reaches a minimum, increases to a secondary peak, and then decreases as the material overages. Figure 1 shows schematically what is proposed to occur during RRA. It was reported by Cina (References 1-3) that processing to the minimum of the retrogression curve, followed by re-aging, led to the favorable combination of T6 strength and T73 SCC resistance. Wallace et al (Reference 5) investigated the new RRA process and utilized a modified heat treatment at lower retrogression temperatures and longer times. This technique allowed a thicker section to be heat treated than the thin sheet used by Cina. He found that retrogressing for 3-4 hours at 180°C followed by re-aging gave yield strengths of 73 KSI (500 MPa). This is within the T6 range. Both studies found the conductivity to be 38% ICAS which was within the T7 range.

Little is known about the precipitation process occurring during RRA. The original work by Cina (Reference 1-3) indicates that the retrogression process takes place for short times (60 seconds and under) and the minimum hardness is much greater as the temperatures increase from 200°C to 270°C. These observations suggest rapid reaction kinetics. Wallace et al (Reference 5) investigated the temperature range reported by Cina and concluded temperature ranges of 160°C-180°C for 4-6 hours can be used. The original temperature-times required in the patent (Reference 3) are not necessary. It is not clear if the retrogression treatment by Wallace covers the temperature range from 150-270°C. Possibly this range could be substituted for the duplex heat treatment in order to combine the T6 and T7 tempers.

The various precipitation reactions that occur in the 7075 alloys are well documented (9-16). The aging sequence has been generally accepted as the following:

Most investigators have concentrated on the early stages of age hardening involving studies of G.P. zone formation, the structure of G.P. zones, zone kinetics, and the various precipitation reactions during the aging process. Unfortunately, there has not been as much microstructural work performed to examine the characteristics of T6 material (References 9-10) aged for shorter times. There has been an excellent paper analyzing 7075-T6, 7075-T7 and a form of the RRA

(Reference 11). The paper presented data on the various precipitate characteristics: size, line fraction, and density for the various tempers (Reference 11).

The increase in electrical resistivity of 7075 is well established. It follows a sigmoidal curve. The G.P. zones interfere both with the positive and negative carriers (Reference 17). The decrease in resistivity is associated with the precipitation of Zn, Mg and Cu. In 7075 and 7091 the solid state reactions are related to thermodynamic and kinetic parameters. A change in the time or temperature will change the kind and volume fraction of precipitators (n' and n). The lowest resistivity occurs when the non-coherent n precipate forms in the overaged condition. The decrease in resistivity is related to the type and amount of precipitates present. It is an Arrhenius relationship.

Conductivity is a rapid way of determining the temper of a 7000 series aluminum and a conductivity > 37% ICAS indicates SCC resistance, although this relationship is not proven. The microstructural features affect the conductivity and strength; therefore, conductivities should be considered in conjunction with hardness and yield strength.

The partly coherent phase, n', makes the largest contribution to hardening; the G.P. zones make the second largest. The incoherent phase, n, makes no contribution to hardening (Reference 11).

The recommended treatment to convert 7075 T6 to the T73 temper is to overage the T6 material at 177°C for 8 hours (Reference 8). This is within the temperature range reported by Wallace (Reference 5). Re-aging this modified T73 should give results similar to RRA. The objectives of the present study are the following:

- (1) to explore the use of longer times and lower temperatures that might allow heat treatment of thick sections.
- (2) to explore the use of T6 as a starting temper.

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- (3) to apply the RRA treatment to 7075 and 7091 alloys.
- (4) to establish hardness and conductivity profiles for retrogression and re-aging reactions.
- (5) to study the correlation between conductivity and hardness.
- (6) to establish if RRA conditions can be attained starting with T73 tempers.

### **EXPERIMENTAL PROCEDURE AND RESULTS**

Two 7000 Series aluminum alloys, 7075 and 7091, were selected for this retrogressing and reaging study. The T6 condition of 7075 and 7091, and T651 condition of 7075 were subjected to the RRA study. Specimens consisted of small rectangular pieces of 7075,  $1" \times 1/2" \times 3/8"$ , (25.4 x 12.7 x 9.5 mm) and circular pieces of 7091 rod,  $3/8" \times 1/2"$  diam, (9.5 mm x 12.7 mm diam.) The T6 heat treatment is given in Table 1. The compositions of 7075 and 7091 are reported in Table 11.

Retrogression was accomplished by "up quenching" in a silicone oil bath followed by a cold water quench. The time for the center of the specimen to reach temperature was calibrated using suitably instrumented test samples. The retrogression times were measured after the specimens had reached the bath temperature.

Hardness and electric conductivity measurements were made after cold water quenching. The hardness was taken on a standard Rockwell hardness tester, B scale. Conductivity measurements were made with a magna flux RM 150 eddy current meter which gives conductivity in percentage of the international annealed copper standard (% IACS).

The retrogressed specimens were then re-aged by placing them in an air oven set at 121°C for 24 hours. Hardness and conductivity measurements were taken after re-aging.

A series of experiments were conducted to evaluate the quenching ability of the silicone fluid. Test coupons with cemented thermocouples in the center were quenched from 480°C. The times to reach ambient and intermediate temperatures were recorded. These results are shown in Figures 2-3.

Aging experiments were conducted on 7075 and 7091 in order to establish the normal aging response of these alloys. Coupons were solution treated at 480°C for 2 hours, cold water quenched, and immediately aged: the aging times extended from 1 hour to 3 days, the aging temperatures were 50°C, 120°C, 160°C, 200°C and 240°C. A minimum of 3 specimens were tested at each condition.

### **TEST SPECIMENS**

The tensile coupons were taken from the edge and center location of plate segments in both the T651 and RRA tempers. The tensile specimens were made in accordance with ASTM standards (Reference 18). The overall size was 2" (50.8 mm) x 0.160 (4.06 mm) diam, with a 0.75" (19.05 mm) gauge length. The tensile tests were performed on a Tinius Olsen machine at a strain rate of .03 in/in/min (0.76 mm/mm/min). The tensile data is shown in Tables 3-6. Table 5 is a summary of the hardness and conductivity of the material used for the tensile specimens. Table 6 gives the data for the material used in the exfoliation test.

### **EXFOLIATION TESTS**

The exfoliation corrosion susceptibility tests were carried out in accordance with ASTM a34-79. (Reference 19). This method provides an exfoliation test for the 7xxx series aluminum alloys that involves continuous immersion in a solution containing 4M sodium chloride, 0.5 M potassium nitrate, and 0.1 M nitric acid at  $77 \pm 5^{\circ}F$  ( $25 \pm 3^{\circ}C$ ). The solution is diluted to 1 liter of distilled or deionized water. A modified version of the test method was used on test coupons that were 1" (25.4 mm) cube. The test is a very severe test. Hence the specimens were removed from the

solution periodically, washed, dried, and visually examined. 7075-T651, RRA and 7075-T73 conditions were examined. The results demonstrate varying degrees of exfoliation or none at all. The hardness and conductivity values for the specimens used in the ex co tests are given in Table 6.

### CONVENTIONAL AGING

Alloys 7075 and 7091 were solution heat treated, quenched, and aged. Hardness and conductivity values were plotted as a function of time for 160°C and 200°C (Figure 4 and 5). Maximum hardness was retained at 200°C for less than 100 minutes, whereas at 160°C it took 300 hours for a noticeable decrease.

Conductivity remained constant at 160°C for approximately 100 minutes; however, at 200°C conductivity starts to increase in less than 20 minutes.

### QUENCHING RATE

Specimens were quenched from solution heat treat temperatures in water and oil. The temperature versus time profiles are shown in Figure 2. As illustrated by the curves, silicone oil is a considerably less effective quenchent than water.

Specimens were up quenced in silicone oil from room temperature (to several retrogression temperatures). The temperature versus time profiles for up quenching are shown in Figure 3. It can be seen that the specimen reaches the bath temperature in approximately two minutes; therefore, retrogression time was measured starting two minutes after up quenching.

### RETROGRESSION AND RE-AGING

Electrical conductivity measurements indicate that during retrogression conductivity increases. During re-aging a further increase of about 0.5% IACS is found. Conductivity increases as the precipitate coherency decreases. The conductivity is lowest with the maximum G.P. zone and highest in the overaged state. The as received 7075 T651 plate had electrical conductivity of 33% IACS. The laboratory blank had a conductivity of 41% IACS.

Given the premise that the higher stress corrosion cracking (SCC) resistance is attributed to the reversion of the coherent phase and the formation of a noncoherent phase (overaging), it is suggested that the increase in conductivity correlates with an increase in SCC resistance. Conductivities between 33% IACS and 42% IACS indicate microstructural features ranging from coherent to semi-coherent to noncoherent precipitates.

The data indicates that there is an incubation time during which no change in conductivity is observed. At 160°C the incubation time is approximately 40 minutes, however at 200°C and above no incubation time was observed. The conductivity results are shown in Figures 6-10.

### **HARDNESS**

The effect of retrogression treatment on hardness for 7075-T651 was determined at 240°C. 220°C, 200°C, 185°C, 180°C and 160°C. The 7091-T6 was evaluated at all temperatures except 160°C and 180°C. Additional 7075 specimens were reheat treated as a T6 temper and

evaluated at 220°C and 200°C. Hardness measurements showed a difference between the T6 and T651 temper. Figures 11-16 show the results for the retrogression treatment as well as the re-aging at 120°C for 24 hours.

The general shape of the hardness curves are similar to the schematic representation showed by Figure 1. The data shows the higher retrogression temperatures produce a lower initial hardness minimum at shorter times. Plotting log time vs 1/T results in the linear curve shown in Figure 17. Re-aging causes the hardness to be restored. It was observed that re-aging also increased the conductivity for the retrogressed material. This implies that the resistance to SCC should also improve. The T73 SCC resistance temper has a conductivity in the range of 38-40% ICAS.

Figures 18 and 19 show that at retrogression temperatures of 160°C and 180°C that the time at temperature can be extended to several minutes. This implies that the RRA treatment should be applicable to thick aluminum plate.

### **TENSILE PROPERTIES**

Substandard size specimens were used to obtain tensile properties. As a result of specimen geometry, yield strengths may not reflect true material properties. Figures 20 & 21 shows the location and size of the subsize specimens.

The yield strengths appear to be low in all the tests and in some cases couldn't be obtained because of the extensiometer response. The requirements for the gauge length to diameter ratio is 4:1. The subsize specimens in this test were borderline to marginal. Any slight slippage of the extensiometer would result in a variation of the yield strength as measured by the 0.2% off set. The ultimate strengths tended to be lower, but the results are consistent.

The results for the usual as received 7075-T651 plate is 78 KSI (540 MPa) yield strength, 87 KSI (600 MPa) ultimate strength and 10-14% elongation. The 7075-T73 results are 60 KSI (414 MPa) yield strength and 70 KSI (483 Ma) ultimate strength. Table 3 shows that the specimens from the center are slightly stronger than those from the edge. The important conclusion of the test is that the hardness values are a good representation of the strength and the RRA treatment results in hardness values close to the as received T651.

Table 4 shows high strength levels for 7075 T73 material. The recommended treatment for converting the T6 to T7 appears to be an extension of the retrogression temperature. The estimated hardness from Figure 18 is Rb 79. The re-aging of the T73 raises the strength level. The T73 temper falls within the RRA parameter studied at 160°C. The re-aging of the T73 temper results in a hardness and conductivity increase on the same order of magnitude as the RRA. The conductivity and hardness of the material used in this test are recorded in Table 5.

### **EXFOLIATION RESULTS**

Figures 22 and 23 show the results of the exfoliation tests of the RRA material. Figure 22 indicates that the RRA material was more resistant to exfoliation than the T651. The T651 material started to show evidence of exfoliation within a few hours. Table 23 shows the results of T651, T73 and RRA. The hardness and conductivity of the material used in the test are reported in Table 5. The RRA and T73 have a rating of P according to the ASTM standard a34-79. The 7075 T651 shows exfoliation.

### DISCUSSION

There are several papers (References 9-10) which investigate 7075 in the T6 and T7 conditions. Ardell et al (Reference 11) discuss characteristics of the T6, T7 and a modified RRA temper. The results of these investigations are valuable to the present interpretation of the RRA temper.

### **RETROGRESSION PROCESS**

The minima in the retrogression curve, shown as a schematic representation in Figure 1, have been described by Park & Ardell (Reference 11) to have a minimum precipitate density which is attributed to a G.P. zone dissolution. Based on the premise that the primary source of the T6 strength is the coherent G.P. zone, the drop in yield strength shown by Wallace (Reference 5) and the hardness shown in Figures 11-16 appears to be due to the dissolution of that coherent phase. The concensus of most investigators was that the G.P. solvus temperature was approximately 130°C. Therefore, the G.P. dissolution would be complete at the retrogression temperatures in this investigation. Ungar et al (Reference 12) have shown that at temperatures as high as 160°C the G.P. zone dissolution would never be reached for alloys with both Zn contents greater than 5.5% and Mg contents greater than 1%. The 7075 and 7091 alloys have Zn and Mg contents greater than these values, and based on Ungar's work (Reference 12), we can conclude that the G.P. zone dissolution is prevented and the formation of n' enhanced. This possibility was reported by Delasi and Adler (Reference 10) who found simultaneous G.P. and n' dissolution for temperatures up to 217°C.

Figures 11-16 show that the time required to reach the minimum in the retrogression curve decreases with increasing temperature and the minimas are lower. This is consistent with the concept of faster dissolution kinetics with increasing temperatures. The data fits a plot of the form

$$\ell n (t) = \frac{A}{T} + b \tag{1}$$

where ln(t) = time

A = slope

T = absolute temperature °k

b = intercept

Lorimer and Nicholson (Reference 14) pointed out that during G.P. dissolution, there is a growth of the pre-existing n' and the incubation of new n' particles on the remaining G.P. zones occurring simultaneously. The rapid decrease in the retrogression curves suggest that these reactions do not occur at the same rate. The work of Ardell and Park (Reference 11) shows that the precipitate density starts to increase after the minima is reached and that there is a growth in the n and n' particles.

The rate controlling reaction based on the existing evidence in the literature (Reference 12-16) must be the G.P. dissolution and therefore equation 1 is related to the G.P. dissolution rate.

Figures 11 - 16 show that there is a limit to the initial drop in hardness, and it begins to increase at some point.

The results of Ardell and Park (Reference 11) indicate there is a change in the volume fraction of n' and n after the minimum is reached. Inoue et al (Reference m) suggest that n', a semi-coherent precipitate, provides the greatest contribution to the strength.

The T6 treatment followed a three day room temperature age after a super saturation solution treatment. This 3 day delay allowed the zones or clusters to reach a critical size. These clusters are stable at the higher temperatures. The artificial aging at 120°C dissolves fewer clusters and a finer, more evenly distributed precipitate network is obtained with higher properties.

As a general rule, the lower temperatures allow a greater number of nuclei to form than are formed at higher temperatures. Increased exposure time at lower temperature allows a greater number of nuclei to become stable for lower growth. Higher aging temperatures result in a smaller number of nuclei and, thus, each precipitate particle grows to a larger size.

The results of Ardell and Park (Reference 11) indicate that after the minimum in the retrogression curve, there is a noticeable increase in the volume fraction along with a gradual increase in precipitate size. The volume fraction is made up of n' and n precipitates. Their results indicate that at longer times there is an increase in the rate of precipitate growth. There is also a corresponding increase in the volume fraction. This growth is overaging where the incoherent particle n is predominant. There is a drop in strength and hardness.

The plots of hardness vs time at temperatures above 185°C shows a sharper minima and then increases to a secondary peak and then decreases. At temperatures of 185°C and below the secondary maximum remains constant for long periods of time before decreasing. This is attributed to a slower nucleation and growth rate.

The electrical conductivity results are consistent with these microstructural features. The incubation period shown in Figures 6-9 can be attributed to the G.P. dissolution. The conductivity does not change appreciably until the G.P. dissolution is no longer the controlling mechanism. It has been reported (Reference 17) that G.P. zones interfere with positive and negative carriers and causes a much lower conductivity than the other precipitates. The linear increase of the sigmoidal curve can be attributed to a mix of n' and n particles. The upper limit of the sigmoidal curve is attributed to the non-coherent n phase. This behavior is consistent with the reaction kinetics for changes of electrical resistivity as related to precipitation changes, i.e. — the resistivity decreases with the increase in the non-coherent phase.

### **RE-AGING**

The re-aging of retrogressed material improved the hardness. (See Figures 11-16 and Tables 5 and 6.) Ardell (Reference 11) showed an increase of n' during aging. Figures 11-16 show that reaging improves the hardness of retrogressed material even after long periods of time when coarsening of the precipitate was thought to take place.

The RRA treatment is a two step aging process: step 1 is retrogression and step 2 is re-aging. In the initial step of retrogression there is partial dissolution of the preexisting G.P. zones, which may not be complete (Reference m). The remaining G.P. zones act as nucleation sites for n' particles (References n-p). The dissolution of G.P. zones enriches the matrix in Zn and Mg which in turn promote the nucleation and growth of n' (MgZn<sub>2</sub>). In the re-aging process both G.P. and n' zones can nucleate and grow. However, the nucleation and growth of n' is dominant over that of the nucleation and growth of the G.P. zones (References 13-17) because the stability of n' is much greater than G.P. zones.

For long retrogression times the increase in strength/hardness on re-aging is due to the high solute concentration in the matrix. A higher solute concentration will allow for the nucleation and growth of n'. The experimental results for the T73 material, heated at 160°C for 24 hours then re-aged and heated at 180°C for 8 hours then re-aged (Table 4), support this rationale.

The various changes that take place in the hardness Figures 11-16 and yield strength (Reference 5) is represented schematically in Figure 1. This representation of the retrogression curve can be represented by 3 zones which are as follows:

- Zone 1 drop in hardness/strengths to a minimum value
- Zone 2 increase in hardness/strength to a maximum
- Zone 3 decrease in hardness/strength. This decrease is slower at lower temperatures.

Zone 1 is the RRA process described by Cina (References 1-3). This is the G.P. zone reversion. Zones 2 and 3 are the nucleation and growth of n' and n. The longer retrogression times in Zone 3 is overaging. Based on Figure 1 we can interrupt the various forms of the T7 temper as Zone 3; especially those that are a duplex treatment or start with the T6 initial condition. There are T73 tempers that have good corrosion resistant properties and a 10% loss in strength. These probably fall close to the maximum on the retrogression curve in Zone 3. By re-aging these tempers we can regain most of the T6 strength.

Based on these results it should be possible to heat treat thick sections by heating the center to a minimum on the retrogressed curve. This puts the surface time/temperature and the maximum in Zone 3. When the material is re-aged the final strength level is uniform or shouldn't vary more than 2-3 ksi (13.8 MPa - 20.7 MPa). Calculations based on the cooling/heating rate data indicate than an infinite slab 1" thick will reach the 150-200°C temperature range in 1 hour.

### CONCLUSIONS

- 1 The initial rapid drop in hardness is a 1st order reaction rate in which the G.P. zone dissolution is thought to be the rate controlling process.
- 2 Retrogression of the T6 temper at 180°C for 6 hours and re-aging appears to give the optimum combination of hardness and conductivity.
- 3 Some of the suggested T73 tempers are only a longer time at a retrogression temperature. Reaging improves the hardness/strength and is the equivalent of an RRA treatment.
- 4 The incubation period on the 185 + 160°C conductivity plots are thought to be related to G.P. zone depletions. The increase in conductivity is attributed to n' and n formation. The maximum conductivities at very long times at a retrogression temperature is over-aging.
- 5 The high conductivity (upper T73) range at 160°C can be obtained while maintaining the T6 hardness. Some of the T6 hardness can be regained by re-aging T73 tempers.
- 6 There is no linear relationship between conductivity and hardness. The hardness/strength must be considered in conjunction with conductivity.
- 7 Based on hardness and conductivity test 7091 responds more readily to RRA than 7075.

### **REFERENCES**

- B. Cina and B. Ranish: "New Techniques for Reducing Susceptibility to Stress Corrosion of High Strength Aluminum Alloy," Israel Aircraft Industries Ltd; Israel, Published Work, 1976.
- 2 B. Cina: <u>International Conference on Aluminum Industrial Products Proceedings.</u> ASM. Pittsburgh, PA, October 1974.
- 3 United States Patent 3856584, 24 December 1974.
- 4 R. S. Kaneko, Metal Progress, 1980, Vol 117, No. 5, P. 41.
- 5 W. Wallace, J. C. Dedde and Mc DeMalherbe, "A New Approach to the Problem of Stress Corrosion Cracking in 7075-T6 Aluminum," National Aeronautical Establishment Report Ottowa, Ontario, K1AOR6, Sept., 1981.
- 6 Military Specification: Heat Treatment of Aluminum Alloys, MIL-H-6088, February 1971.
- 7 <u>Aluminum Standards and Data 1979</u> The Aluminum Association of America, Sixth Edition, 1979, P. 146.
- 8 Alcoa, Green Letter on 7075-T73 1971, Aluminum Company of America Aerospace Structure Metals Handbook, MPDC, Vol. 3, Code 3207, Columbus, Ohio, December 1980.
- 9 P. N. Adler, R. Delasi and G. Geshwind, Metall Trans., 1972, Vol 3, P. 3191.
- 10 R. Delasi and P. N. Adler, Met Trans. A, 1977, Vol. 8A, P. 1177.
- 11 A. J. Ardell and J. K. Park, "Fundamental Investigation of the Retrogression and Re-aging (RRA) of Commercial Al. base Alloys and Its Effects on Stress Corrosion Cracking," UCLA School of Engineering and Applied Sciences, Contract No. N00014-81-K-0292, May 1982.
- 12 T. Ungar, J. Lendval and I. Kovacs: Aluminum, 1979, Vol. 55, P. 663.
- 13 H. Inoue, T. Sato, Y. Kojima and T. Tukahashi, Met Trans A. 1981, Vol. 12A, P. 1429.
- 14 G. W. Lorimer and R. B. Nicholson: Acta Met, 1966, Vol. 14, P. 1009.
- 15 J. Ajonnes and Chr. T. Siemsen: Acta Met, 1970, Vol. 18, P. 881.
- 16 I. Kovacs, J. Lendval, T. Ungar, G. Groma, and J. Lakner: Acta Met. 1980, Vol. 28, P. 1621.

- 17 J. Smugeresky, H. Herman and S. R. Pollock, Acta Met, 1969, Vol. 7, P. 803.
- 18 Metals Physical Mechanical, Corrosion Testing, ASTM, E 8, Part 10, 1981, P. 210.
- 19 ANSI/ASTM, G3-1, "Standard Method of Test for Exfoliation Corrosion Susceptibility in 7xxx Series Copper Containing Aluminum Alloys (Exco Test)," 1982, Annual Book of ASTM Standards, Part 10, P. 981.

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TABLE 1. HEAT TREATMENT OPTIONS FOR 7075

T6: Solution Treat, Quench, and Age 120°C/79 hours to Peak Strength.

T6: Solution Treat, Quench and 2 Stage Age (95°C/4 hours and 155°C/8 hours).

T73: Solution Treat, Quench and 2 Stage Over-Age (105°C/8 hour + 165°C/24 hours)

to Improve Stress Corrosion Resistance.

T76: Solution Treat, Quench and 2 Stage Over-Age (100°C/8 hours + 165°C/24 hours).

T7351: Start With T651 + Over-age at 162°C for 24-30 Hours.

TABLE 2. COMPOSITION LIMITS OF ALUMINUM ALLOYS USED

### **WEIGHT PERCENT**

Element	<u>7091</u>	<u>7075</u>
Cu	1.50	1.2 2.0
Mg	2.74	2.1 – 2.9
Zn	5.96	5.1 – 6.1
Ti	<del>-</del>	0.2
Cr	0.0013	0.18 - 0.28
Mn	0.0018	0.3
Si	0.055	0.4
Ti	-	0.2
AI	Balance	Balance
Со	0.50	

TABLE 3. TENSILE PROPERTIES OF 7075 ALUMINUM ALLOY PLATE

Temper	Location	0.2% Offset Yield Strength KSI (MPa)	Tensile Strength KSI (MPa)	Elongation ::: 25.4 mm (1 in)
7075 T-651 As Received	Center	-	78.5 (542)	12.5
7075 T-651 As Received	Тор	-	81.2 (560)	12.5
*7075 T-651 + RRA	Center	67.3 (464)	80.3 (554)	12.5
*7075 T-651 + RRA	Тор	_	76.3 (527)	12.5

<sup>\*</sup>Retrogressed at 180°C for 6 hours and re-aged at 120°C for 24 hours.

TABLE 4. TENSILE PROPERTIES OF 7075 ALUMINUM ALLOYS AT VARIOUS TREATMENTS

ldent. No.	Temper	0.2% Off Set Yield Strength KSI (MPa)	Ultimate Tensile Strength KSI (MPa)	Elongation in 25.4 mm (1 in)
1	7075-T7351 (T651 plus 24 hrs at 162°C)	65.8 (464)	78.8 (544)	12,5
2	7075-T7351 + Re-aged at 120°C 24 hrs	67.4 (465)	79.9 (551)	12.5
3	7075-T651 Plus Retrogression 180°C for 6 hrs	80.7 (557)	81.3 (560)	12.5
4	7075-T651 + Retrogression at 180°C for 6 hrs and re-aged for 24 hrs at 120°C	70.6 (487)	82 (566)	12.5
	QQ-A-250/12E T651	68 (469) Minimum	78 (538) Minimum	7 (minimum)
	Alcoa Green Letter T73	57 (363) Minimum	69 (476) Minimum	7 (minimum)

TABLE 5. HARDNESS AND CONDUCTIVITY % I.A.C.S. FOR THE VARIOUS THERMAL TREATMENTS GIVEN THE 7075 TENSILE TEST IN TABLE 3

Ident. No.	Description of Treatment		ogression for Aged Conductivity % I.A.C.S.		aged at for 24 hrs Conductivity % I.A.C.S.
1	T-7351 Temp (Start with T651 Age at 162°C for 24 hrs	81	40	_	_
2	T-7351 Plus Re-aging for 24 hrs at 120°C	81	40	84	41
3	Retrogressed at 180°C for 24 hrs	85	38	-	_
4	Retrogressed at 180°C for 6 hrs Plus Re-aged at 120°C for 24 hrs	86	38	88	39

TABLE 6. 7075 SPECIMENS HEAT TREATED AND USED IN EXPOLIATION TEST (EXCO).

ldent. No.		Retrogressed and/or Aged		Re-aged at 120°C for 24 hrs	
		Hardness R <sub>B</sub>	Conductivity % 1.A.C.C.	Hardness R <sub>B</sub>	Conductivity % I.A.C.S.
1	T73 Temper and Re-aged for 24 hrs.	82	40	86	41
2	T73 Temper	82	40	-	
5	7075 Plate T651 Temper	90	33	-	_
14	7075 Plate T651 Temper	90	33	_	_
23	Retrogressed at 200°C for 10 min.	86	37	89	38
24	Retrogressed at 200°C for 30 min.	81	40	85	41

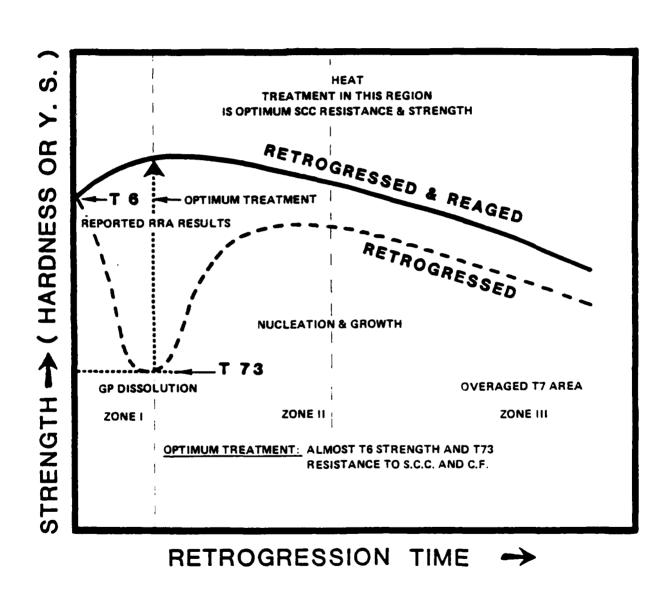
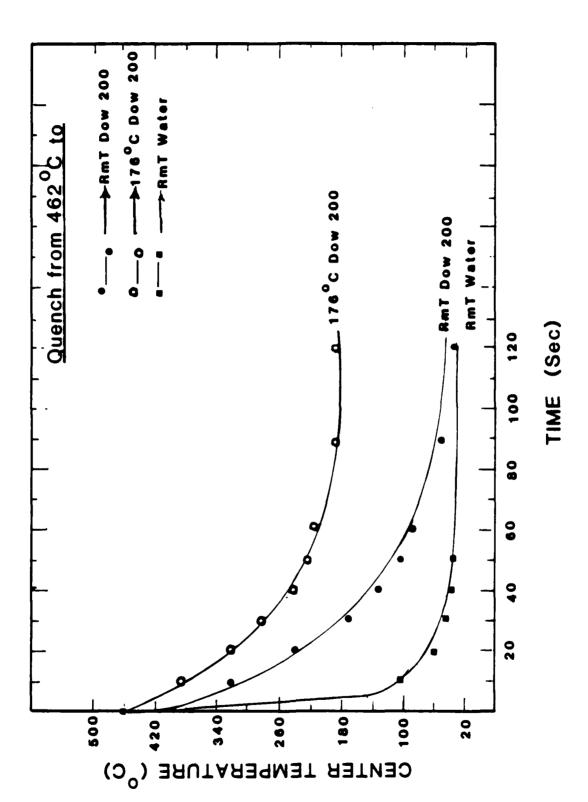


Figure 1. Changes in hardness during retrogression and re-aging. The optimum treatment is shown by the dotted line. The material is retrogressed to the minimum hardness and re-aged.



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Figure 2. Temperature vs. time. Profile for 7091 quenched from the solution treatment temperature to room temperature and an intermediate temperature.

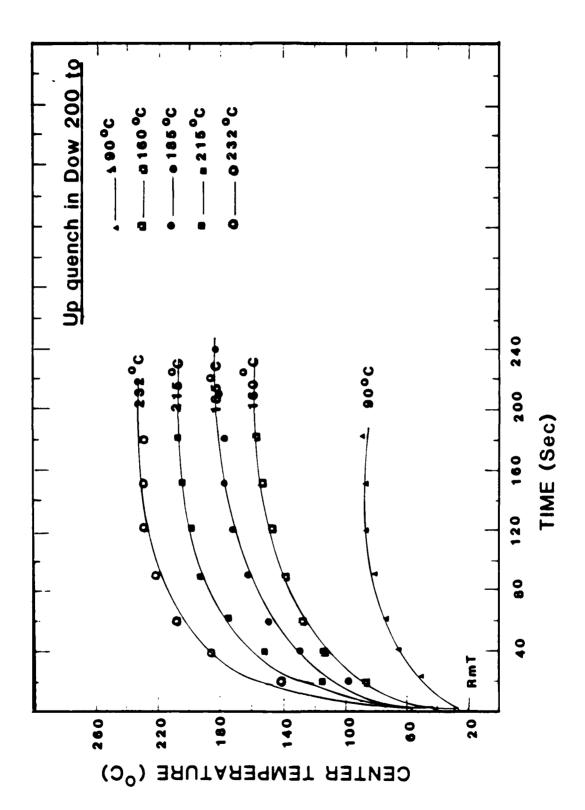


Figure 3. Temperature vs. time profiles for 7091 up quenched to various temperatures from room temperature

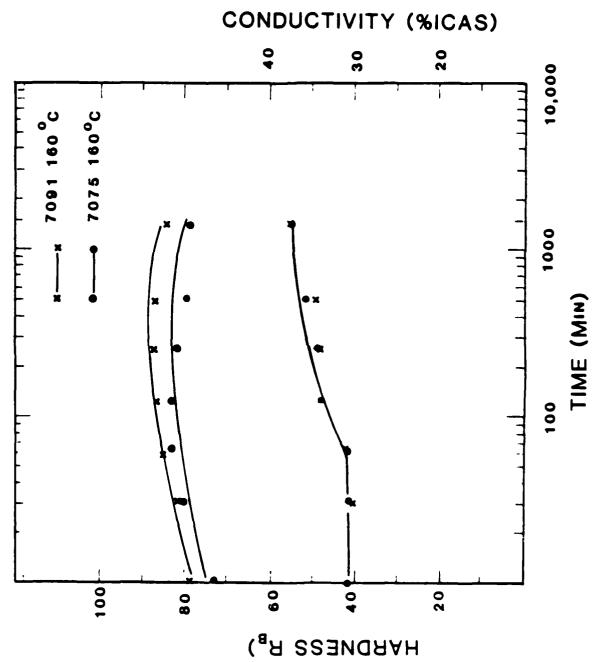


Figure 4. Hardness and conductivity results obtained for 7091 and 7075 alloys solution treated, quenched and held at 160°C for various periods of time.

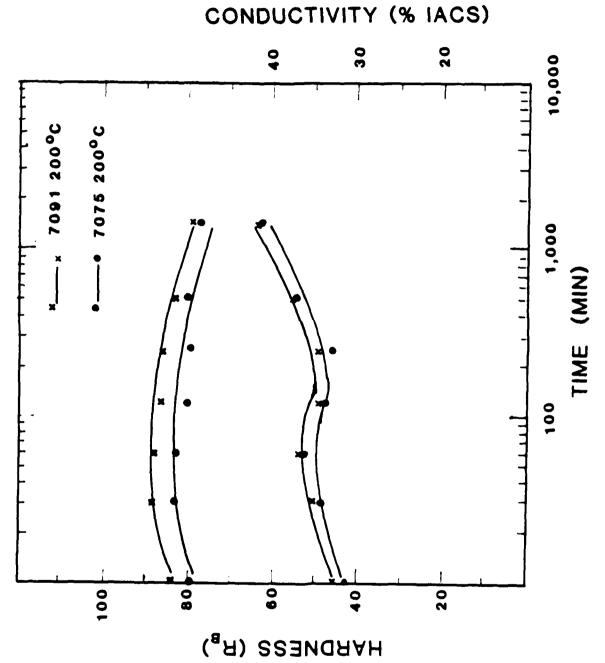


Figure 5. Hardness and conductivity results obtained for 7091 and 7075 alloys, solution treated, quenched and held at 200°C for various periods of time.

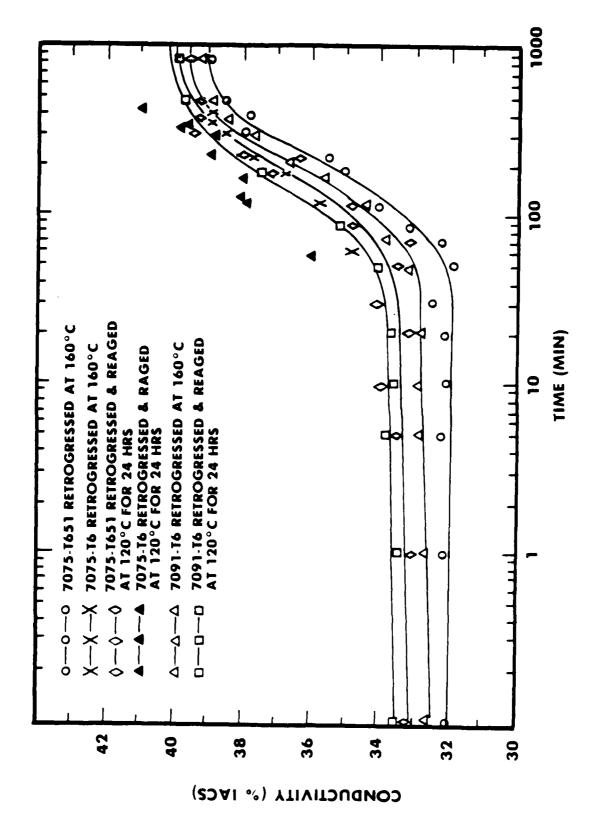


Figure 6. Electrical conductivity of 7075 T651 and 7091-T6 retrogressed at 160°C for up to 1440 minutes and re aged.

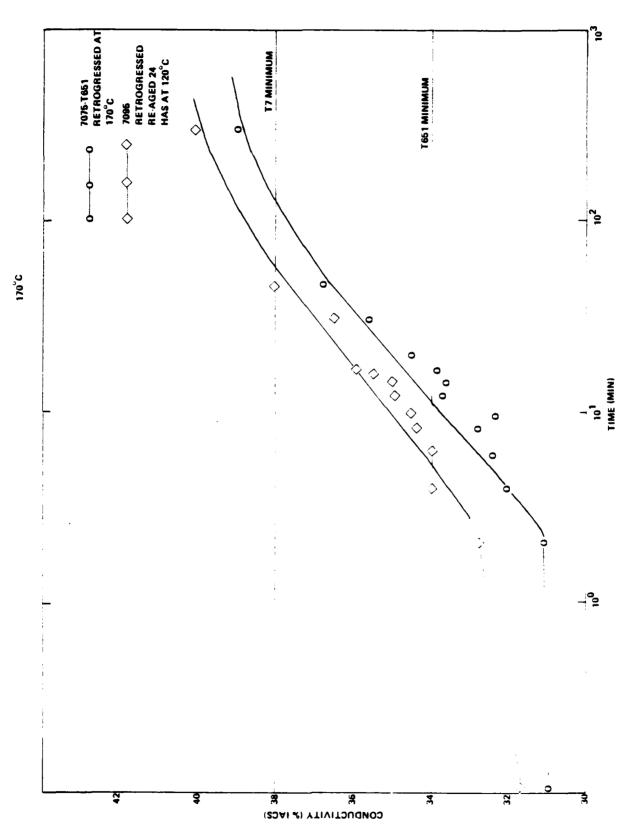


Figure 7. Electrical Conductivity at 7075-T651 retrogressed at 170°C for up to 300 minutes and re aged.

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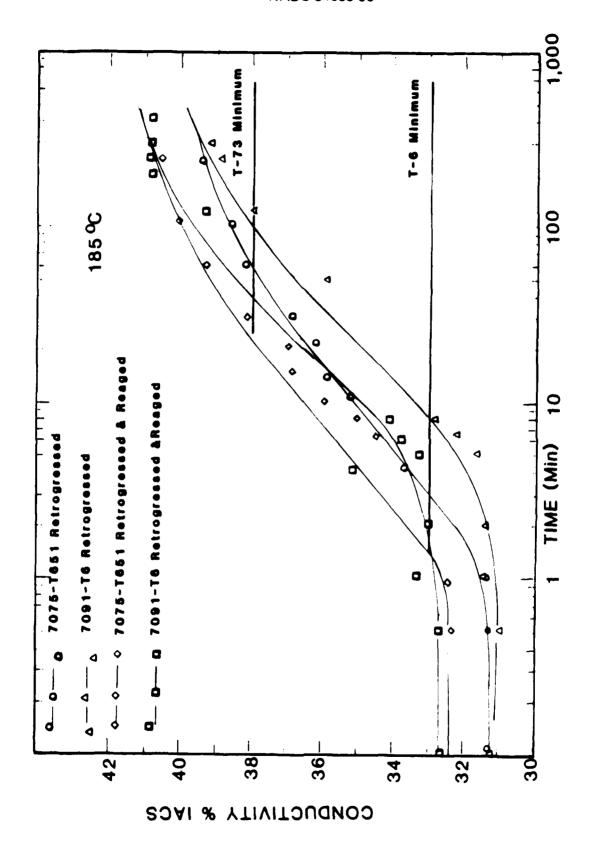


Figure 8. Electrical conductivity of 7075 T651 and 7091-T6 retrogressed at 185°C for up to 240 minutes and re-aged.

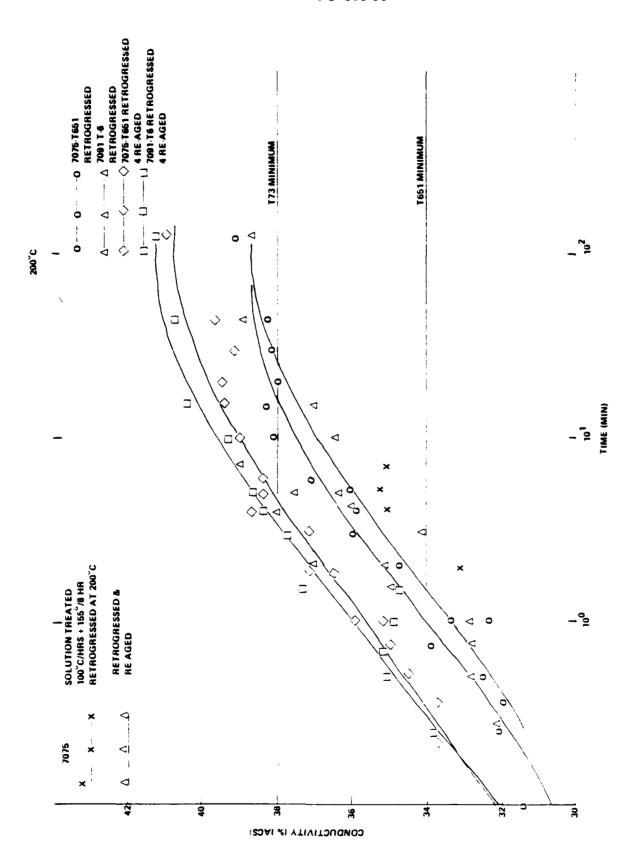


Figure 9. Electrical conductivity of 7075 T651 and 7091-T6 retrogressed at 200°C for up to 100 minutes and re-aged.

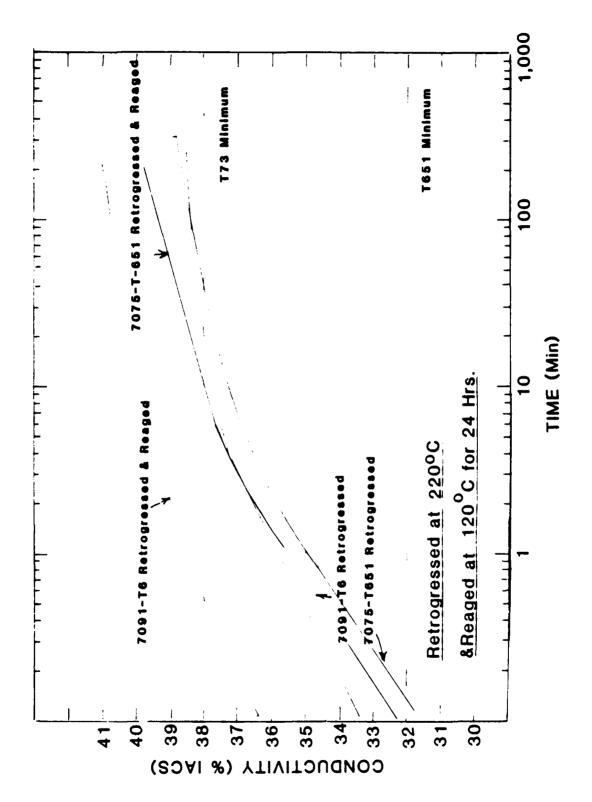


Figure 10. Electrical conductivity of 7075 T651 and 7091 T6 retrogressed at 220°C for up to 100 minutes and re-aged.

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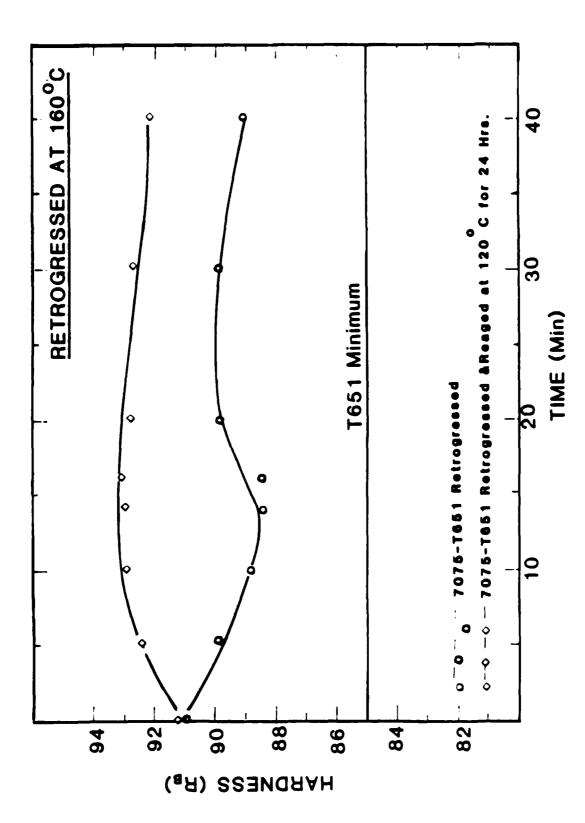


Figure 11. Hardness  $R_{
m B}$  of 7075-T651 retrogressed at 160°C for up to 40 minutes and re-aged.

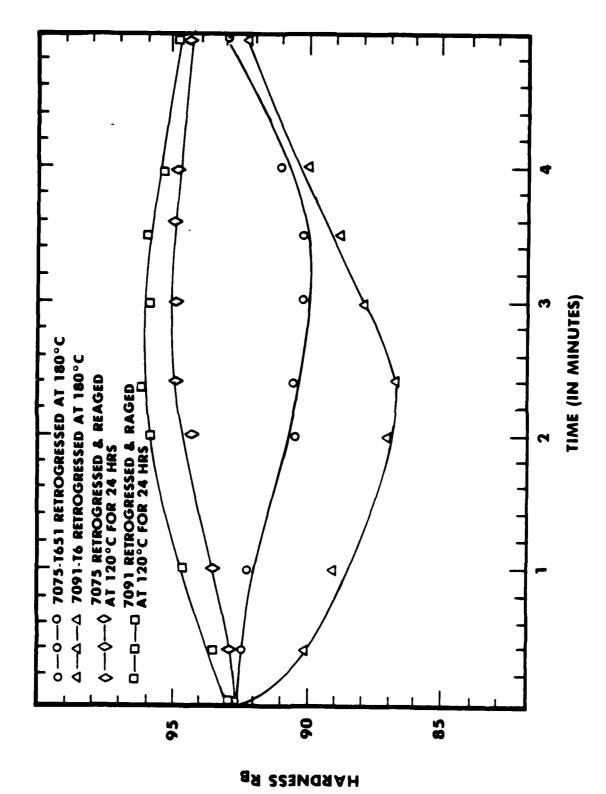


Figure 12. Hardness of 7075 T651 and 7091 T6 retrogressed at 180°C for up to 40 minutes and re-aged.

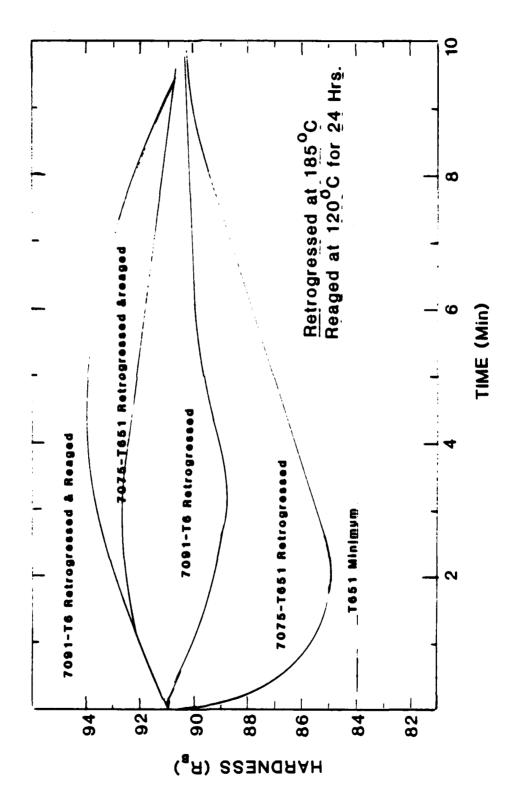


Figure 13. Hardness of 7075 T651 and 7091 T6 retrogressed at 185°C for up to 40 minutes and re-aged.

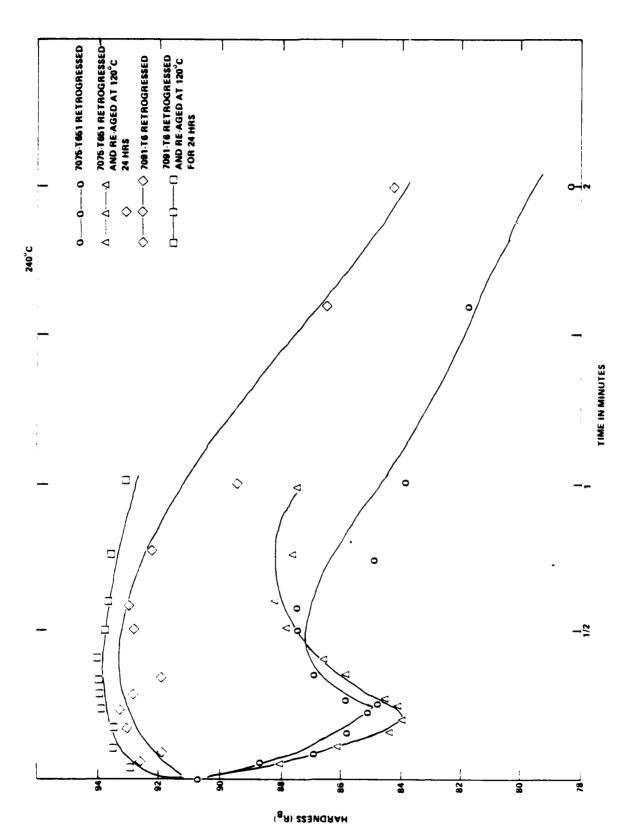


Figure 14. Hardness of 7075 T651 and 7091 T6 retrogressed at 200°C for up to 4 minutes and re-aged.

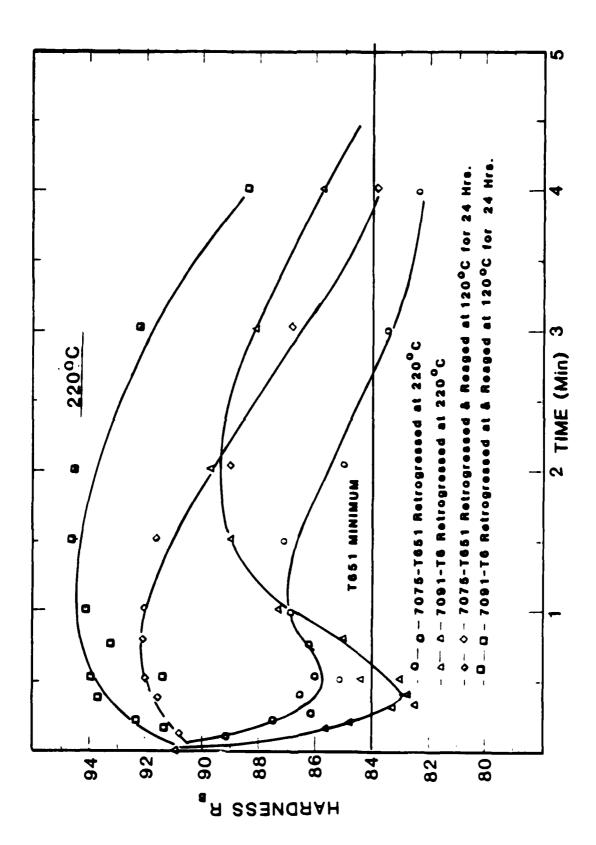


Figure 15. Hardness of 7075 T651 and 7091 T6 retrogressed at 220°C for up to 4 minutes and re aged.

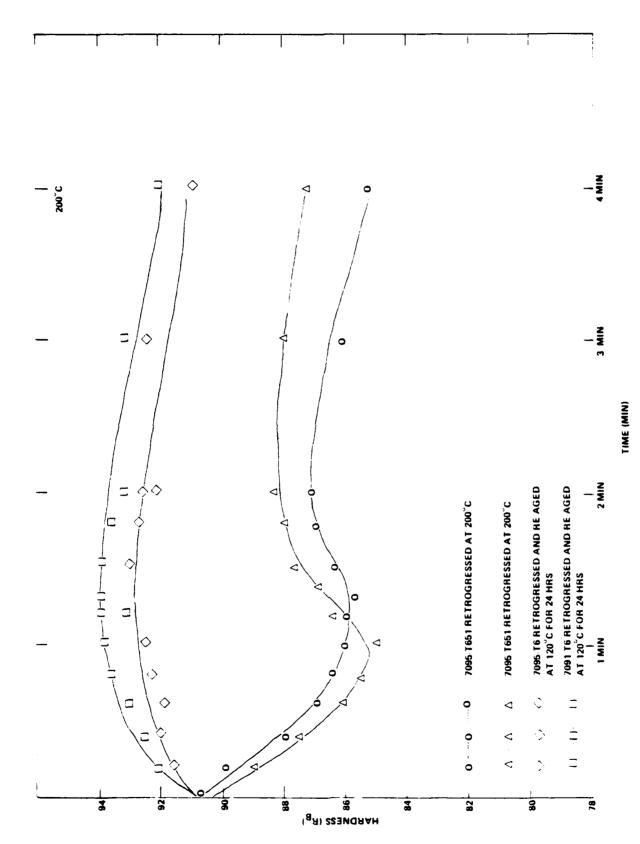


Figure 16. Hardness of 7075 T651 and 7091-T6 retrogressed at 240°C for up to 2 minutes and re-aged.

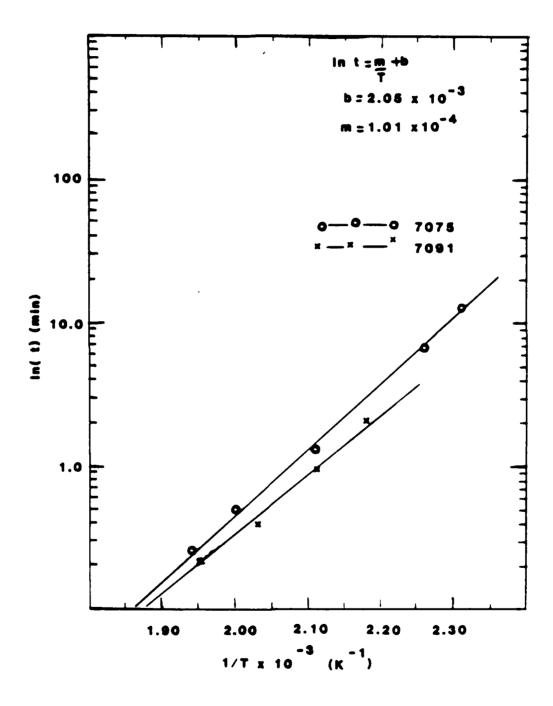


Figure 17. Time to reach minimum hardness vs. i/T where T is  $^{\circ}$  Kelvin. Note curve can be described in  $\ell$ n r = A/T + b.

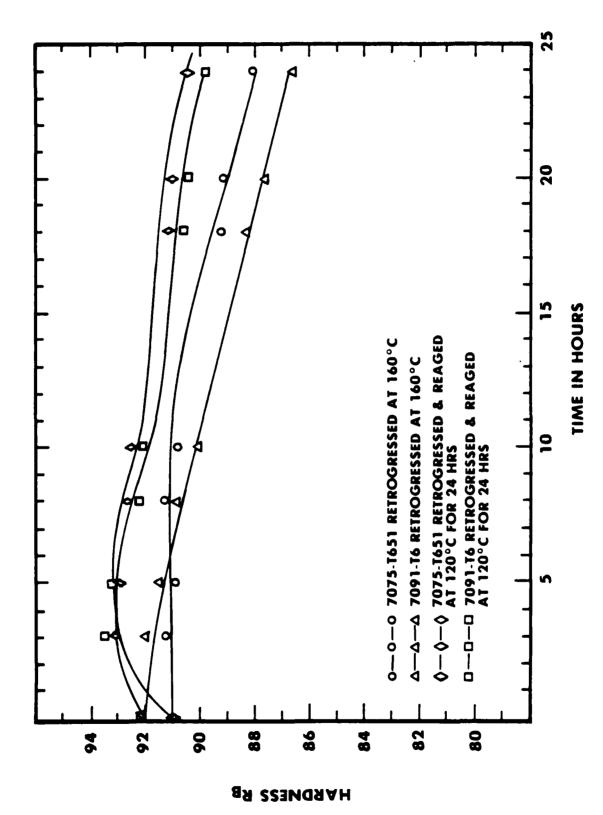


Figure 18. Hardness of 7075 T651 and 7091-T6 retrogressed at 160°C for up to 24 hrs and re-aged.

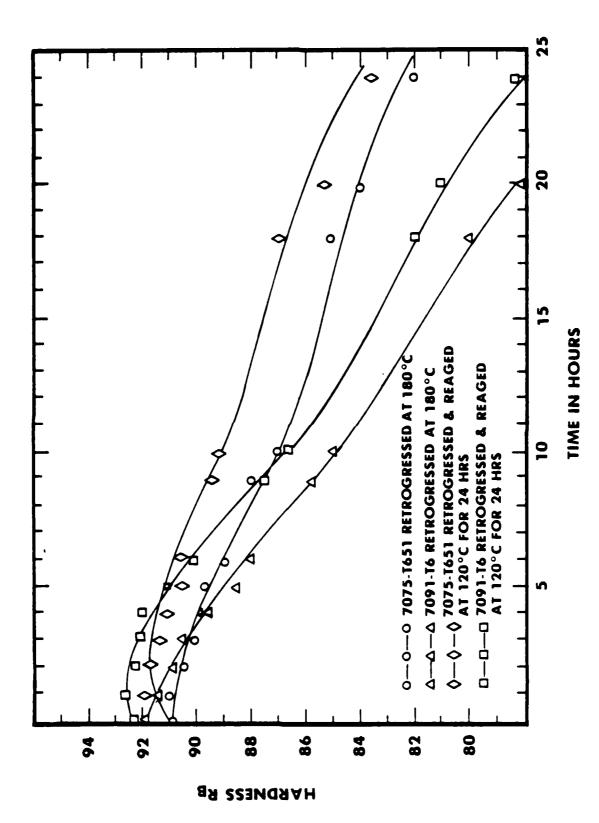
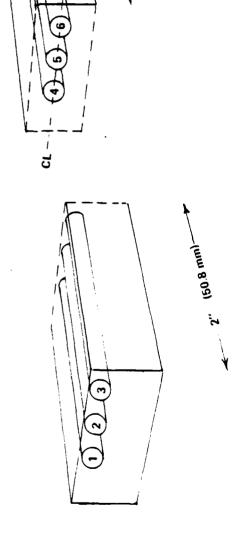


Figure 19. Hardness of 7073 T651 and 7091 T6 retrogressed at 180°C for up to 24 hrs and re-aged.

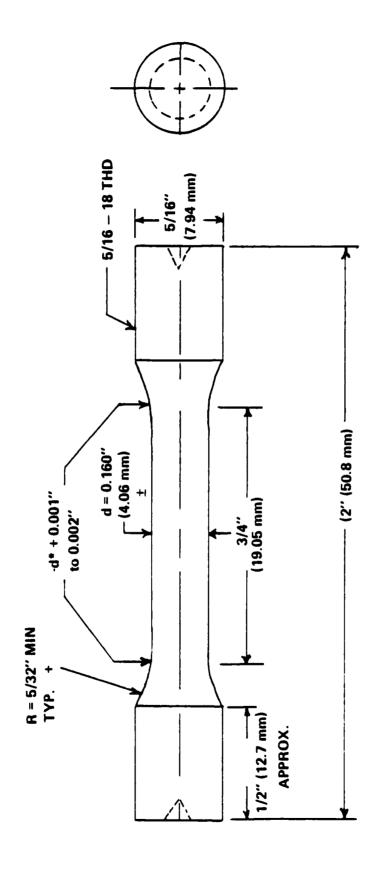
-Z' (50.88 m)

7075-T6 STANDARD HEAT TREATMENT 6 SPECIMENS: 3 FROM PLATE SURFACE (#1, 2, 3) 3 FROM PLATE CL (#4, 5, 6)



Representation of the areas where the subsize tensile blanks were taken from in the as received 7075-T651 and the RRA treatment at 180°C. Figure 20.

### TENSION TEST SPECIMEN

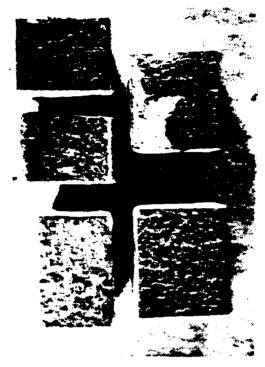


# \*GRADUAL TAPER TO "d" AT CENTER OF SPECIMEN GAGE LENGTH

Figure 21. Tensile specimen dimension taken from ASTM E8. Part 10.



**21 HRS** 



27 HRS 25 MIN



**48 HRS** 

Figure 22. Photographs of the exfoliation test of 7075-T651 and RRA (180°C) at various time intervals during the test.

## EXFOLIATION - EXCO TEST 7075 ALUMINUM

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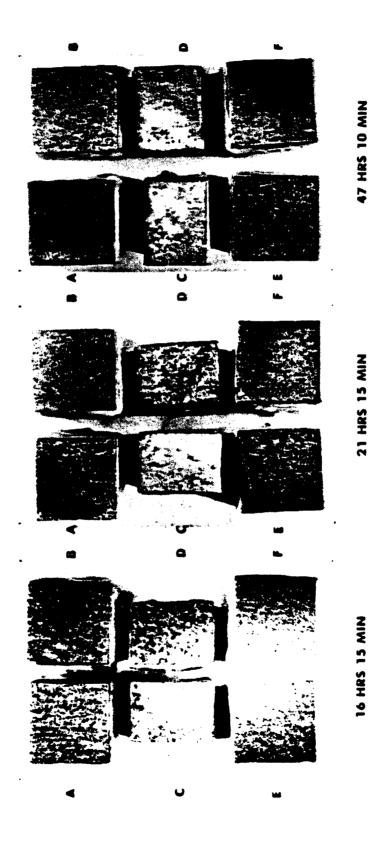


Figure 23. Photographs of the exfoliation (EXCO) tests of 7075-T651, T73 and RRA (200°C) at various time intervals during the test.

C - 7651 D - 7651

- 17351 - 17351

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